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ADVISORY COUNCIL

SUBCOMMITTEE ON MANUAL OF PROCEDURES

Wednesday
February 8, 1978
1:00 P.M.

939 Ellis Street
San Francisco
California 94109

# M I N U T E S

COMMITTEE MEMBERS PRESENT: Lowell V. Smith, Chairperson; Shirley J. Campbell, Joseph D. Coons, Hiram Wolch.

COMMITTEE MEMBERS ABSENT: John L. Nicholson.

STAFF MEMBERS PRESENT: Steve Balestrieri, Dario Levaggi, Walter Oyung, Meg Sanders.

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Chairperson Smith called the meeting to order at 1:15 P.M.

## APPROVAL OF MINUTES OF JANUARY 20, 1978

Mrs. Campbell requested a correction on page 2, paragraph 4, line 5, to read "sampling loop..." and a correction on page 3, LAB 11, 2.1, to read, "Buret, 0-50 ml"

The Committee discussed procedures for editorial consistency.

Mrs. Campbell moved that the Minutes be approved as corrected; seconded by Mr. Coons; carried.

## REVIEW OF LABORATORY METHODS INVOLVING NITROGEN COMPOUNDS

Chairperson Smith asked if comments in the letter from Pacific Gas and Electric Company, dated January 12, 1978, had been addressed in Staff corrections of the Laboratory Methods. Mr. Levaggi replied that the suggestions had been incorporated for discussion at this meeting.

Mr. Levaggi reviewed the procedures as contained in LAB 1. The following corrections were made:

Page 1 - Title to read, "DETERMINATION OF AMMONIA IN EFFLUENTS TAKEN FROM GAS STREAMS"

4.1 - line 1, to read "...brought to 5.0 ml..."

4.1 - line 6, to read"...From the standard curve, determine..."

5.1 - line 1, to read"...Weigh out..."

The Subcommittee discussed the procedure. Dr. Wolch questioned the inconsistency of abbreviations for "gram". Mr. Levaggi agreed that the entire LAB Methods Manual should be revised to change all abbreviations for "gram" to "g".

The Subcommittee made the following changes:

3.2 - line 1, to read,"...'Colorimetric Methods'..."

4.1 - line 2, to read, "N HCl and containing..."

5.1 - line 1, to read,"... .3144 g"

5.1 - line 3, to read"..Dilute this standard solution 1:5..."

5.1 - line 5, to read"..add respectively 0, 1.0, 2.0, 4.0, 5.0 ml..."

5.1 - line 6, to read"..To each tube add..."

6. -"ppm" to be capitalized

LAB 7A, "DETERMINATION OF OXIDES OF NITROGEN IN EFFLUENTS (ALKALINE PERMANAGANATE PROCEDURE)" The Subcommittee discussed the procedure and Mr. Levaggi made the following corrections:

Title to read, "...(ALKALINE PERMANAGANATE PROCEDURE)"

1. - delete line 7

2.2 - to read "50 ml graduated Erlenmeyer Flasks with screw caps"

Add to procedure 2. "APPARATUS" the following:

2.4 - 25 ml graduated test tubes

2.5 - Burrell wrist Action Shaker

3.2 - to read"..Dissolve 80 g of oxalic acid..."

3.3 - line 1, to read"..Add 22.4 ml of concentrated..."

3.3 - line 2, to read, "sulfuric acid to 500 ml of distilled water and dilute to liter."

4.1 - line 6, to read"...thereby requiring..."

4.2 - line 2, to read"...A reagent blank of 2.0 ml..."

4.3 - line 5, to read"...and dilute to the 30 ml mark ..."

4.4 - line 3 and 4, to read"..and transfer 25 ml to a graduated..."

5.1 - line 1, to read, "...and dissolve 0.2198 grams of..."

The Subcommittee made the following additional changes:

4.1 - line 2, to read, "Prepare an aliquot not exceeding 5.0 ml and containing 10-100 ugm of nitrite for analysis."

4.1 - line 4 and 5, delete "The aliquot should not exceed 5.0 ml."
4.4 - line 7, to read 13 mm cuvette,setting..."
5.1 - line 3, to read, "...Dilute 1:5 to..."

LAB 7B - DETERMINATION OF OXIDES OF NITROGEN IN EFFLUENTS (GRAB SAMPLE TECHNIQUE). Mr. Levaggi made the following corrections;

Title - to read,"..(GRAB SAMPLE PROCEDURE)"

2.3 - to read, "Hot plate."

3.1 - line 4 to read, "...cover the flask..."

line 5 to read, "watch glass and heat for..."

Add to Procedure 3. "REAGENTS" the following:

3.4 pH paper, 7-14 pH range.

4.2 - delete line 3, add "Use pH paper to varify a solution of 10 or greater."

4.3 - line 2, to read, "...to a 50 ml volumetric flask..."

4.8 - line 3, to read, "...100% transmittance. The..."

5.1 - line 2, to read, "...dissolve 0.5495 g of potassium nitrate..."

line 5, to read, "this stock solution 1:10 to obtain..."

The Subcommittee reviewed LAB 7B procedure and made the following additional corrections:

3.1 - add to line 3, "This solution is stable for at least two years."

4.8 - line 4, to read, "...determine ugm of $NO_2$ equivalent..."

6. - CALCULATIONS, line 5, to read, "Flask pressure = (760 ml - Hg manometer reading)"

7. REFERENCES, add the following:

7.3 - Federal Register, Vol. 42, No. 160, Thursday, August 18, 1977, Revisions to Reference Methods 1-8, Method 7 pgs.41784-41786.

LAB 14, DETERMINATION OF TRIMETHYLAMINE IN EFFLUENTS. Mr. Levaggi described the procedures and discussed the following changes:

2.1.1- line 5, to read, "...15 ml/min hydrogen, 250-300 ml air, and a temperature of 75° C.

2.2 - deleted

2.3 - changed to 2.2

2.4 - changed to 2.3

3. REAGENTS, the following to be added:

"3.3 Litmus paper, red"

The Subcommittee added the following changes:

1. PRINCIPLE, line 1, to read, "The trimethylamine is absorbed..."

2.1 - line 1, to read, "The gas chromatographic unit..."

2.1.1 - line 1, to read, "A glass 4' x 1/8" analytical column"

4.1 - line 1, to read "...volume of the impinger liquid..."

4.2 - line 1, to read, "Inject 2.0 ul into the injection port..."

5.1 - line 2, to read, "add 0.1691 g ..."

line 3, to read "This standard solution contains..."

6. CALCULATION, line 1, to read, "...trimethylamine in the impinger'

Mr. Levaggi made the following changes for LAB 19, "STANDARDIZATION OF NITRIC OXIDE CALIBRATION GAS":

Title - to read "STANDARDIZATION"

2. - line 5, delete "sensitive"

3.1 - to read, "Greenberg Smith impingers fitted with semi-fine frits. Frits are available from Ace Glass Company, Vineland, New Jersey; Cat. #7201-04."

Renumber 3., 3.1, 3.2, 3.3, 3.4; to read, 4., 4.1, 4.2, 4.3, and 4.4, respectively.

4.1 - line 1, to read, "Absorbing solution."

4.2 - line 1, to read, "Oxalic Acid solution."

4.3 - line 1, to read, "Sulfuric Acid (0.8N). Add 22.4 ml..."

4.4 - line 1, to read, "Nitrate Reagents. Nitra Ver III..."

5.2 - line 1, to read, "Record the original wet test meter reading ($M_1$). By means of the controlling..."

line 6, to read, "As a guide, 100 ppm nitric oxide requires..."

5.3 - line 2, to read, "Record the final wet test meter reading ($M_2$), and remove the impingers from the sampling train."

Add - 5.4 - At least two determinations are to be made and sampling results must agree to within ± 5% of mean.

Mr. Levaggi advised the Subcommittee that the Staff will prepare steps to be added to procedure for 5.4; prepare a sampling train illustration; and prepare a sentence describing the illustration for the Subcommittee's approval.

The Subcommittee discussed the procedure as incorporated in LAB 19 and made the following additional corrections:

2. - line 3, to read, "formed nitrite and nitrate ions thus formed are determined ..."

3.5 - to read, "Hot plate."

4.3 - line 2, to read, "sulfuric acid to 500 ml of distilled water."

4.4 - line 2, to read, "Ver VI (Cat. no. 14119) are available from..."

5. - line 3, to read, "absorbing solution (4.1). The impingers..."

LAB 8, DETERMINATION OF PHENOLS IN EFFLUENTS - Mr. Levaggi made the following corrections:

1.1 - line 3, to read, "by acid, and the aqueous solution is analyzed for phenols by gas"

Reverse order of procedures 2. and 3. Change numbering of each.

2.1 - line 1, to read, "A gas chromatograph is equipped..."

line 2, to read "...fitted with a 7' x 1/8" stainless steel column is packed"

line 3, to read, "with 10% Carbowax 20M/terphthalic acid on acid-washed Chromosorb W"

4.1 - line 1, to read, "All samples are to be..."

line 2, to read, "...within three days, proceed..."

line 3, to read, "...than three days, add..."

4.2 - line 1, to read, "If the sample is contaminated with... heavy oil an extraction"

line 2, to read, "...If the sample is "clean","

4.3 - line 3, to read, "...Cover the tube with..."

line 4, to read, "Check to insure the sample is acidic with litmus paper."

4.4 - line 1, to read, "Inject a 2 ul sample into the gas chromatograph."

line 2, to read, "The operating para-"

4.5 - line 3, to read, "...Comparison of samples may also be made based on the"

line 4, to read, "integrated area technique."

5.1 - line 1, to read, "Weigh 0.1 to 0.11 g ($\pm$0.0001 g) each of reagent grade"

line 4, to read, "...each phenol."

5.2 - line 1, to read, "Dilute standard solution ..."

line 2, to read, "...approximately 25 ugm per ml of each phenol. This"

6. - Calculation 1), to read, "Total ugm of a Phenol* = ..."

Calculation 2), to read, "PPM of a given Phenol = ..."

At Mr. Coons' request Mr. Levaggi agreed to make the necessary changes for the Trimethylamine operating parameters of the chromatograph to follow the form as incorporated in LAB 8, 4.4, as well as to use symbols for certain abbreviations in both Calculations of LAB 8, (6.) and list immediately below the calculations the symbols' definitions.

LAB 15, STANDARDIZATION OF CARBON DIOXIDE CALIBRATION GAS.

Mr. Levaggi made the following corrections:

2. - line 1, to read, "The carbon dioxide gas is absorbed by..."

3.5- to read, "Two Stage Regulator, with a..."

5. - line 1, to read, "Assemble a sampling train..."

5.7- to read, "At least two determinations are to be made and results must agree to within $\pm$ 5% of the mean.

Mr. Levaggi indicated that the above changes for 5.7 will be incorporated in the remainder of the manual. He also told the Subcommittee that the diagram of the Sampling Train for Gravimetric Determination of $CO_2$ will be rescaled.

The Committee made the following changes for LAB 15:

3.2 - to read "Schwartz Absorption Combustion Tubes. These tubes are available from Kontes Glass Co.(Cat. No. K 891250). The tubes..."

4.3 - Drierite (8 to 14 mesh)

LAB 16 - STANDARDIZATION OF CARBON MONOXIDE CALIBRATION GAS

Mr. Levaggi made the following changes:

2. - line 2, to read, "by use of a Hopcalite catalyst. The carbon dioxide thus formed is..."

line 3, to read, "determined gravimetrically after absorption by Ascarite. Any water..."

3.2- to read, "Rotameter (0 to 1 liter range)

3.5- to read, "Schwartz Absorption Combustion Tubes. These tubes are available from Kontes Glass Co. (Cat. No. K 891250). The tubes are filled with the appropriate material and plugged at each end with glass wool."

3.7- line 1 to read, "Hopcalite U-trap. This is constructed..."

line 2, to read, "silicate glass and is approximately..."

line 4, to read, "with glass wool. This trap should be fitted"

line 5, to read, "...assembling and disassembling."

4.4- to read, "Drierite (8 to 14 mesh)

5.1- line 1, to read, "Assemble a sampling train..."

line 3, to read, "...A Hopcalite trap immersed in a boiling water bath; a Drierite trap to..."

5.2 - line 4, to read, "...Calibration gas to pass through at approximately 0.5 l"

5.5 - line 3, to read "...approximately 0.5 l per"

line 5, to read "The sampling time will depend on the concentration of the carbon monoxide..."

line 6, to read "...in the cylinder. The gain in weight of the Ascarite-Anhydrone tube should be at least 20 mg. As a guide, 200 ppm of carbon monoxide requires a sample size of approximately 60 liters."

6. line 1 to read, "1 mg carbon monoxide per liter is equal to 873 ppm"

7.2 - to read, "Methods of Air Sampling and Analysis, Second Edition, Katz, M., Ed., Intersociety Committee, American Public Health Assn. Washington, D.C. 1977.

Figure 1. - to read, "Heated Hopcalite (Boiling Water Bath)"

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The Subcommittee made the following changes:

5.3 - line 3, to read, "Record the weight (W1).

5.7 - line 1, to read, "At least two determinations are to be made and the results must agree to within ± 5% of the mean.

TIME AND PLACE OF NEXT MEETING

Wednesday, February 22, 1978, 1:30 P.M., 939 Ellis Street, San Francisco, California.

The meeting was adjourned at 3:45 P.M.

Respectfully submitted,

Mabel Harder
Secretary

MH:ms



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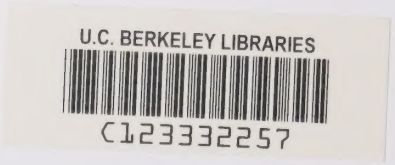


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